Reference (13)

DECLARATION

I, Maho KASEKI, c/o the Inoue & Associates of 3rd Floor, Akasaka Habitation Building, 3-5, Akasaka 1-chome, Minato-ku, Tokyo, Japan do solemnly and sincerely declare that I am conversant with the Japanese and English languages and that I have executed with the best of my ability this partial translation into English of Unexamined Japanese Patent Application Laid-Open Specification No. 2003-131463 and believe that the translation is true and correct.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

December 11, 2004
(Date)

Maho KASEKI

Partial English Translation of Unexamined Japanese Patent Application Laid-Open Specification No. 2003-131463

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(54) [Title of the Invention] Semiconductive belt

(2) At page 2, left column, lines 1 to 13:

[Scope of Claims for Patent]

[Claim 1] A semiconductive belt comprising a polyimide resin having incorporated therein carbon black, wherein said carbon black has a maximum particle diameter of from 1.0 to 1.5 μm as measured with respect to secondary particles of said carbon black, and wherein the standard deviation of the particle size distribution of said secondary particles is 0.5 μm or less.

[Claim 2] The semiconductive belt according to claim 1, which has a carbon black content of from 8 % by weight or more, based on the weight of said polyimide resin in terms of the weight of the solids contained therein, and which has a tensile modulus of 6.0 MPa or more as measured in accordance with JIS K7127.

[Claim 3] The semiconductive belt according to claim 1 or 2, wherein the common logarithm of the surface resistivity (log (Ω/□)) of said semiconductive belt is 9 or more.

<u>(3) At page 5, left column, line 32 to page 6, upper right column, 4th line below Table 1:</u>

[0035]

[Examples] Hereinbelow, the present invention will be described with reference to the following Examples which illustrate the construction and effects of the present invention.

[0036] Example 1

Into 1,800 g of N-methyl-2-pyrrolidone (NMP) was dispersed 84 g of dry carbon black "MA-100" (trade name; manufactured and sold by Mitsubishi Chemical Corporation; the primary particle diameter: 22 nm) (which is an amount such that the carbon black (CB) content of the polyimide becomes 18.7 % by weight, based on the weight of the polyimide in terms of the weight of the solids contained therein) while stirring by a ball mill at room temperature for a predetermined period of time, to thereby obtain a carbon black dispersion in NMP. Into the dispersion were dissolved 294 g of 3,3',4,4'-benzophenonetetracarboxylic acid dianhydride (BPDA) and 108 g of p-phenylenediamine (PDA), and the resultant solution was stirred at room temperature for 3 hours in nitrogen atmosphere, to thereby perform a reaction to obtain a polyamide acid solution having a viscosity of 1,000 poise.

[0037] The obtained polyamide acid solution was applied onto an inner wall of a mold having an inner diameter of 330 mm and a length of 500 mm by using a dispenser, to thereby form a coating having a thickness of 400 μm, followed by rotating

of the mold at a rate of 1,500 rpm for 10 minutes to render uniform the thickness of the coating. After blowing a hot air having a temperature of 60 °C onto the mold from outside thereof for 30 minutes while rotating the mold at a rate of 250 rpm, the mold was heated at 150 °C for 60 minutes, and the heating was continued while elevating the temperature to 300 °C at a rate of 2 °C/min, followed by further heating of the mold at 300 °C for 30 minutes, thereby removing the solvent and the water produced by dehydration-cyclization reaction of the polyamide acid from the coating and performing an imide-conversion of the polyamide acid present in the coating. Subsequently, the mold was allowed to cool to room temperature, followed by peeling-off of the coating from the mold, to thereby obtain a seamless semiconductive belt having a thickness of 74 to 76 μm.

[0038] Example 2

A seamless semiconductive belt having a thickness of 74 to 76 μm was obtained in substantially the same manner as in Example 1, except that the carbon black was changed to "Printex V" (trade name; manufactured and sold by Degussa Huls AG; the primary particle diameter: 25 nm) and that the conditions for stirring by the ball mill during the preparation of the carbon black dispersion in NMP were adjusted so as to control

the maximum particle diameter of the secondary particles of carbon black present in the polyamide acid solution and the standard deviation of the particle size distribution of the secondary particles to their respective values as shown in Table 1.

[0039] Comparative Example 1

A seamless semiconductive belt having a thickness of 74 to 76 μm was obtained in substantially the same manner as in Example 1, except that the conditions for stirring by the ball mill during the preparation of the carbon black dispersion in NMP were adjusted so as to control the maximum particle diameter of the secondary particles of carbon black present in the polyamide acid solution and the standard deviation of the particle size distribution of the secondary particles to their respective values as shown in Table 1.

[0040] Comparative Example 2

A seamless semiconductive belt having a thickness of 74 to 76 μm was obtained in substantially the same manner as in Example 1, except that the carbon black dispersion in NMP obtained in Comparative Example 1 was used, and that the CB content of the polyimide was adjusted to 7.4 % by weight, based on the weight of the polyimide in terms of the weight of the solids contained therein, so that the common logarithm

of the surface resistivity (log (Ω/□)) of the semiconductive belt became 13.5 ± 0.5.

[0041] Comparative Example 3

A seamless semiconductive belt having a thickness of 74 to 76 μm was obtained in substantially the same manner as in Example 1, except that the conditions for stirring by the ball mill during the preparation of the carbon black dispersion in NMP were adjusted so as to control the maximum particle diameter of the secondary particles of carbon black present in the polyamide acid solution and the standard deviation of the particle size distribution of the secondary particles to their respective values as shown in Table 1.

[0042] Tests for various evaluations

With respect to the secondary particles of carbon black in the polyamide acid solution, the standard deviation of the particle size distribution and the maximum particle diameter were measured using a centrifugal particle size analyzer "SA-CP3" (trade name; manufactured and sold by Shimadzu Corporation). The tensile modulus of the semiconductive belt was obtained in accordance with JIS K7127 as follows. A test specimen having a size of 10 mm × 250 mm was cut out from the semiconductive belt. The test specimen was subjected to a tensile test using a tensilon under conditions wherein the

distance between the chuck grips was 150 mm, and the pulling rate was 1 mm/min. From the resultant data, the tensile modulus of the semiconductor belt was calculated. The surface resistivity of the semiconductive belt was measured by using Hiresta-UP (trade name; manufactured and sold by Mitsubishi Chemical Corporation) equipped with a UR-100 probe. In the measurement, the voltage applied was 500 V, and the surface resistivity was determined, based on the current as measured 10 seconds after the start of the application of electricity. With respect to the durability of the semiconductive belt, a durability test was performed in accordance with JIS P8115 by using an MIT folding endurance tester and a test specimen having a width of 15 mm and a length of 110 mm, under the load of 1 kg. The durability of the semiconductive belt was evaluated in accordance with the following relative criteria: ○ (very good), △ (good) and × (poor), based on the frequency of application of load until the test specimen is broken.

[0043] The results are shown in Table 1 below.

[0044]

[Table 1]

	Ex. 1	Ex. 2	Comp. Ex. 1	Comp. Ex. 2	Comp. Ex. 3
Maximum particle diameter (μm)	1.05 0.36	1.10 0.38	1.75 0.55	1.75 0.55	0.8 0.18

Standard deviation (μm) CB content (wt %)	18.7	18.0	18.7	7.4	20.6
Logarithm of surface resistivity	13.2	12.8	8.1	13.1	13.1
Tensile Modulus (MPa)	6.5	6.3	6.5	5.9	6.8
Durability	○	○	○	○	×

As is shown in Table 1 above, the semiconductive belts obtained in Examples 1 and 2 not only exhibit the desired surface resistivity, but also exhibit improved tensile modulus or durability as compared to that of the semiconductive belt obtained in Comparative Example 2 or 3. Further, as is apparent from the contrast between Example 1 and Comparative Example 1, in the case where the carbon black present in the polyimide resin has too wide a range of secondary particle diameter distribution and too large a maximum particle diameter of the secondary particles, the semiconductive belt produced from such a polyimide resin poses a problem in that the carbon black markedly increase the conductivity of the semiconductive belt and, hence, the surface resistivity of the semiconductive belt (e.g., intermediate transfer belt used in an electrophotographic recording device) is markedly lowered.